

**PATENT ABSTRACTS OF JAPAN****REFERENCES  
RE REJECTION**(11)Publication number : **08-073724****JUN 13 2003**(43)Date of publication of application : **19.03.1996**

(51)Int. Cl.

**C08L 69/00****C08G 64/04****C08K 5/10**(21)Application number : **06-234475**(71)Applicant : **NIPPON G II PLAST KK**(22)Date of filing : **05.09.1994**(72)Inventor : **ISAWA KENICHI  
ITOI HIDEYUKI****(54) POLYCARBONATE RESIN COMPOSITION**

(57)Abstract:

**PURPOSE:** To provide a polycarbonate resin composition excellent in mold release properties, heat resistance and transferability and suitable for optical application such as an optical disc.

**CONSTITUTION:** This optical composition contains 100 pts.wt. aromatic polycarbonate resin having a terminal hydroxyl group content of 2-40mol% and a molecular weight distribution (Mw/Mn) of 2.0 to 2.8 as determined by gel permeation chromatography, and 0.01-0.1 pt.wt. partial ester of an aliphatic carboxylic acid and a polyhydric alcohol.

**LEGAL STATUS**[Date of request for examination] **19.03.2001**

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number] **3394336**[Date of registration] **31.01.2003**

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

[Date of extinction of right]

Copyright (C); 1998,2003 Japan Patent Office



\* NOTICES \*

Japan Patent Office is not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. \*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

---

DETAILED DESCRIPTION

---

[Detailed Description of the Invention]

[0001]

[Industrial Application] this invention relates to the polycarbonate (PC may be called hereafter) resin constituent excellent in the moldability suitable for manufacturing mold goods for optics, such as an optical disk, a lens, and prism, thermal resistance, etc.

[0002]

[Description of the Prior Art] PC resin has been conventionally used for an optical use, for example, an optical disk, an information disk, a lens, prism, etc. widely as a material excellent in transparency, thermal resistance, and mechanical strength. And in connection with it, improvement of thermal stability or a mold-release characteristic has come to be called for strongly.

[0003] If its mold release resistance is strong in case an optical disk, an information disk, a lens, etc. are released from mold from metal mold since they are manufactured mainly by injection molding, they will produce the warp of mold goods and will cause optical strain. So, use of a release agent is required in the case of fabrication. It is required that the irregularity of 1 micrometer or less of a stamper should especially be correctly imprinted with an optical disk at the time of fabrication. This imprint is greatly influenced with the fluidity of a resin, and the performance of a release agent. Furthermore, since injection molding of an optical disk is usually performed at the elevated temperature of 300-400 degrees C, the thermal stability of a resin is required similarly.

[0004] Conventionally, as a release agent of PC resin, paraffin, a silicone oil, a fatty acid, fatty acid ester, fatty-acid partial ester, etc. are known. In order to improve the mold-release characteristic of PC resin for optical disks especially, fatty-acid partial ester, especially the partial ester of polyhydric alcohol are used. A fatty-acid monoglyceride is mentioned as partial ester of polyhydric alcohol.

[0005] For example, invention which carries out 0.01-0.2 weight section addition of the saturated fatty acid monoglyceride of C16-C22 to the PC resin 100 weight section into PC resin for the purpose of improvement of a mold imprint of the mold goods for optics is indicated by JP,2-48081,B. Moreover, invention which carries out 0.1-5 weight section addition of the aliphatic monoglyceride to the PC resin 100 weight section into PC resin for the purpose of antistatic is indicated by JP,55-4141,B. Furthermore, invention which carries out 0.05-5 weight section addition of the partial ester of a saturation monovalent carboxylic acid and polyhydric alcohol to the PC resin 100 weight section into PC resin for the purpose of improvement in a mold-release characteristic is indicated by JP,47-41092,B.

[0006] In these invention, in order to acquire sufficient mold-release characteristic, it is necessary to add the partial ester of a lot of saturation monovalent carboxylic acids and polyhydric alcohol. However, in the molecular structure, aliphatic partial ester contains the hydroxyl and tends to cause a pyrolysis. So, when aliphatic partial ester is added so much in PC resin, it is burned at the time of fabrication, or has the problem that fault, like a silver streak happens arises. moreover, continuous molding performed in the case of manufacture of an optical disk besides the fault about the thermal stability of the above [ PC resin which contains an aliphatic monoglyceride so much ] -- setting -- metal mold -- the problem that a front face is polluted is produced the above-mentioned thermal stability on the other hand and metal mold -- although the problem of surface contamination is solved by lessening extremely the addition of the aliphatic monoglyceride in PC resin, or the partial ester of polyhydric alcohol, in this case, the mold-releases characteristic from the metal mold of PC resin run short, and, especially in manufactures, such as an optical disk, it has the problem that the advanced imprint of the micron order from a stamper is not obtained

[0007] Moreover, if it is an addition of the same grade as an aliphatic monoglyceride or the partial ester of polyhydric alcohol when using other release agents, for example, the full ester of polyhydric alcohol, the problem about thermal stability will not be produced. However, it has the problem that the advanced imprint of micron order is not enough.

[0008]

[Problem(s) to be Solved by the Invention] The purpose of this invention is offering PC resin constituent excellent in a mold-release characteristic, thermal resistance, and imprint nature, and is offering PC resin constituent suitable for optical uses, such as an optical disk, in more detail.

[0009]

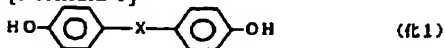
[The means for solving invention] End hydroxyl concentration is 2-40-mol %, and this invention is a polycarbonate resin constituent containing the aromatic polycarbonate resin 100 weight section whose molecular weight distributions (Mw/Mn) measured by the gel permeation chromatography are 2.0-2.8, and the partial ester 0.01 of an aliphatic carboxylic acid and polyhydric alcohol - the 0.1 weight sections. this invention is the above-mentioned polycarbonate resin constituent whose viscosity average molecular weights of the above-mentioned aromatic polycarbonate resin are 12,000-18,000 again. this

✓ invention is the above-mentioned polycarbonate resin constituent whose above-mentioned partial ester is the monoglyceride and/or diglyceride of a saturation monovalent fatty-acid acid of carbon numbers 12-24 again.

[0010] PC resin used for this invention can be preferably obtained by carrying out the melting polymerization of an aromatic dihydroxy compound and the carbonic acid diester to JP,4-175368,A like a publication, although it can obtain by the phosgene method or the melting polymerization method. Especially the above-mentioned aromatic dihydroxy compound is the following formula (\*\* 1), although not limited.

[0011]

[Formula 1]



(Inside of a formula, and X) [0012] <BR>. [Formula 2]



[0013]

[Formula 3]



-O-, -S-, -SO-, or -SO2- it is -- R1 And R2 a hydrogen atom or a monovalent hydrocarbon group -- it is -- a case -- together -- becoming -- a ring -- you may form -- R3 it is a bivalent hydrocarbon group and one or more monovalent hydrocarbon groups or a halogen machine replaces the hydrogen atom of a phenylene group -- you may have -- the compound shown is mentioned the above -- as a monovalent hydrocarbon group, alkyl groups, such as a methyl, ethyl, a propyl, n-butyl, t-butyl, and a pentyl machine, etc. can be mentioned A methylene, an ethylidene, a propylidene machine, etc. can be mentioned as a hydrocarbon group of the above-mentioned bivalency.

[0014] As the above-mentioned aromatic dihydroxy compound, specifically Screw (4-hydroxyphenyl) methane, 1, and 1-screw (4-hydroxyphenyl) ethane, 2 and 2-screw (4-hydroxyphenyl) propane, 2, and 2-screw (4-hydroxyphenyl) butane, 2 and 2-screw (4-hydroxyphenyl) octane, a screw (4-hydroxyphenyl) phenylmethane, 2 and 2-screw (4-hydroxy-3-methylphenyl) propane, 1, and 1-screw (4-hydroxy-3-t-butylphenyl) propane, Screw (hydroxy aryl) alkanes, such as 2 and 2-screw (4-hydroxy-3-BUOMO phenyl) propane, 2, and 2-screw (4-hydroxy-3, 5-dimethylphenyl) propane Screw (hydroxy aryl) cycloalkanes, such as 1 and 1-screw (4-hydroxyphenyl) cyclopentane, 1, and 1-screw (4-hydroxyphenyl) cyclohexane Dihydroxy aryl ether, such as 4 and 4'-dihydroxy diphenyl-ether, 4, and 4'-dihydroxy -3 and the 3'-dimethylphenyl ether, Dihydroxy diaryl sulfides, such as 4 and 4'-dihydroxydiphenyl sulfide, 4, and 4'-dihydroxy -3 and a 3'-dimethyl diphenyl sulfide Dihydroxy diaryl sulfoxides, such as 4 and 4'-dihydroxydiphenyl sulfoxide, 4, and 4'-dihydroxy -3 and a 3'-dimethyl diphenyl sulfoxide Dihydroxy diaryl sulfones, such as 4 and 4'-dihydroxy diphenylsulfone, 4, and 4'-dihydroxy -3 and 3'-dimethyl diphenylsulfone, can be mentioned. Especially 2 and 2-screw (4-hydroxyphenyl) propane (\*\*\*\* when calling it bisphenol A hereafter) is [ among these ] desirable.

[0015] Specifically as the above-mentioned carbonic acid diester, diphenyl carbonate, ditolyl carbonate, screw (chlorophenyl) carbonate, m-cresyl carbonate, dinaphthyl carbonate, screw (diphenyl) carbonate, diethyl carbonate, dimethyl carbonate, dibutyl carbonate, dicyclohexyl carbonate, etc. are mentioned. Especially diphenyl carbonate is [ among these ] desirable.

[0016] the above-mentioned carbonic acid diester -- desirable -- less than [ 50 mol % ] -- you may contain the dicarboxylic acid or dicarboxylic-acid ester of an amount not more than 30 mol % still more preferably As such a dicarboxylic acid or dicarboxylic-acid ester, a terephthalic acid, an isophthalic acid, a sebacic acid, a decanedioic acid, dodecane diacid, terephthalic-acid diphenyl, isophthalic-acid diphenyl, sebacic-acid diphenyl, decanedioic-acid diphenyl, dodecane diacid diphenyl, etc. are mentioned. A polyester polycarbonate is obtained when such a dicarboxylic acid or dicarboxylic-acid ester is used together with a carbonic acid diester.

[0017] It faces manufacturing the polycarbonate used by this invention, and, as for the above-mentioned carbonic acid diester, it is desirable to use more preferably 1.01-1.20-mol 1.01-1.30 mols in the amount of 1.01-1.10 mols to one mol of aromatic dihydroxy compounds. When it desires big end hydroxyl concentration, this mole ratio is made small.

[0018] It faces manufacturing a polycarbonate and an aromatic dihydroxy compound, a carbonic acid diester, and the multifunctional compound that has three or more functional groups in 1 molecule can also be used. As the above-mentioned multifunctional compound, the compound which has a phenolic hydroxyl group or a carboxyl group in [ three or more ] 1 molecule is desirable, and the compound containing especially three phenolic hydroxyl groups is desirable. Specifically 1, 1, and 1-tris (4-hydroxyphenyl) ethane, alpha-methyl - alpha, alpha', and alpha'-tris (4-hydroxyphenyl) - 1, 4-diethylbenzene, alpha, alpha', alpha''-tris (4-hydroxyphenyl) - 1, 3, 5-triisopropyl benzene, A FURORØ glycine, 4, 6-dimethyl - 2, 4, 6-tree (4-hydroxyphenyl)-heptane - 2, 1, 3, 5-TORI (4-hydroxyphenyl) benzene, The 2 and 2-screw -4, 4-(4 and 4'-dihydroxy phenyl) cyclohexyl propane, trimellitic acid, 1 and 3, 5-benzene tricarboxylic acid, pyromellitic acid, etc. can be mentioned. 1, 1, and 1-tris (4-hydroxyphenyl) ethane among these, alpha, alpha', alpha''-tris (4-hydroxyphenyl) - 1, 3, and 5-triisopropyl benzene is

desirable. It is usually desirable to use preferably the 0.03 mols or less of the 0.001-0.02 mols of the above-mentioned multifunctional compounds in the amount of 0.001-0.01 mols still more preferably to one mol of aromatic dihydroxy compounds. [0019] Using an alkaline compound catalyst, the above aromatic dihydroxy compounds, a carbonic acid diester, and if needed, the polycarbonate used for this invention carries out melt polycondensation of the above-mentioned multifunctional compound, and manufactures it. Especially as an alkaline compound, an alkali metal compound and/or an alkaline-earth-metal compound are mentioned.

[0020] As the above-mentioned alkaline compound, the organic acid chloride, an inorganic-acid salt, an oxide, a hydroxide, a hydride, or an alcoholate of metals, such as alkali metal or alkaline earth metal, etc. is desirable. these compounds are independent -- or two or more sorts can be combined and it can use

[0021] As the above-mentioned alkali metal compound, specifically A sodium hydroxide, a potassium hydroxide, a lithium hydroxide, a sodium hydrogencarbonate, A potassium hydrogencarbonate, a carbonic acid hydrogen lithium, a sodium carbonate, potassium carbonate, A lithium carbonate, sodium acetate, potassium acetate, an acetic-acid lithium, a sodium stearate, A stearin acid potassium, a lithium stearate, a sodium borohydride, A lithium borohydride, phenyl-ized boron sodium, a sodium benzoate, A benzoic-acid potassium, a benzoic-acid lithium, disodium hydrogenphosphate, the potassium phosphate, phosphoric-acid hydrogen 2 lithium, the disodium salt of bisphenol A, 2 potassium salt, 2 lithium salt, the sodium salt of a phenol, potassium salt, lithium salt, etc. are mentioned.

[0022] Specifically as the above-mentioned alkaline-earth-metal compound, a calcium hydroxide, a barium hydroxide, a magnesium hydroxide, a strontium hydroxide, a calcium hydrogencarbonate, carbonic acid hydrogen barium, carbonic acid hydrogen magnesium, carbonic acid hydrogen strontium, a calcium carbonate, a barium carbonate, a magnesium carbonate, a strontium carbonate, a calcium acetate, a barium acetate, a magnesium acetate, a strontium acetate, a calcium stearate, a barium stearate, a magnesium stearate, stearin acid strontium, etc. can be mentioned.

[0023] As for the above-mentioned alkali metal compound or an alkaline-earth-metal compound, it is preferably [ to one mol of aromatic dihydroxy compounds / ten - four mols or less ] desirable to use especially 10-7 to 3xten - six mols preferably in the amount of 10-7 to 2xten - six mols still more preferably 10-7 to ten - five mols. While being able to maintain polymerization activity as it is [ above-mentioned ] within the limits, when the amount of an alkali metal compound or an alkaline-earth-metal compound adds the acid compound later mentioned in the amount which does not have a bad influence on the property of a polycarbonate, in the basicity which these compounds show, fully, it can weaken and neutralization or the polycarbonate which was excellent in a hue, thermal resistance, water resistance, and weatherability, and was excellent in prolonged melting stability is obtained. If an alkali metal compound or an alkaline-earth-metal compound exceeds the above-mentioned upper limit, even if it neutralizes the aforementioned alkali by the method of mentioning later, the inclination which becomes difficult [ it / to maintain thermal resistance, water resistance, forming stay stability, etc. on the outstanding level ] will arise.

[0024] In this invention, other basic compounds and boric-acid compounds can be used with the above-mentioned alkaline compound as a catalyst.

[0025] As the above-mentioned basic compound, at an elevated temperature, for example on easily decomposability or an volatile nitrogen-containing compound, and a concrete target Tetramethylammonium hydroxide ( $\text{Me}_4\text{NOH}$ ), tetraethylammonium hydroxide ( $\text{Et}_4\text{NOH}$ ), Alkyls, such as tetrabutylammonium hydroxide ( $\text{Bu}_4\text{NOH}$ ) and trimethyl benzyl ammonium hydroxide ( $\text{Ph-CH}_2(\text{Me})_3\text{NOH}$ ), The ammonium hydronalium oxides which have an aryl, the Al aryl group, etc. The third class amines, such as a trimethylamine, a triethylamine, a dimethyl benzylamine, and a triphenylamine,  $\text{R}_2\text{NH}$  (the inside of a formula, and R -- alkyls, such as a methyl and ethyl, and a phenyl --) aryl groups, such as toluyl one, etc. -- it is -- the second class amines shown and  $\text{RNH}_2$  (among a formula) R -- the above -- being the same -- the primary amine and 2-methyl imidazole which are shown -- Imidazole derivatives, such as 2-phenyl imidazole, or ammonia, Tetramethylammonium borohydride ( $\text{Me}_4\text{NBH}_4$ ), Tetrabutylammonium borohydride ( $\text{Bu}_4\text{NBH}_4$ ), Basic salt, such as tetrabutylammonium tetraphenyl borate ( $\text{Bu}_4\text{NBPh}_4$ ) and tetramethylammonium tetraphenyl borate ( $\text{Me}_4\text{NBPh}_4$ ), etc. can be mentioned. Tetrapod alkylammonium hydroxide, especially the tetrapod alkylammonium hydroxide for electrons with few metal impurities are [ among these ] desirable.

[0026] As the above-mentioned boric-acid compound, a boric acid, the ester of boric acid, etc. can be mentioned. As the ester of boric acid, they are a general formula and  $\text{B(OR)}_n$ . The ester of boric acid shown by  $(\text{OH})_{3-n}(\text{R})_n$  (R is aryl groups, such as alkyl groups, such as a methyl and ethyl, and a phenyl, etc. among a formula, and n is 1, 2, or 3) is mentioned. Specifically as the above-mentioned ester of boric acid, trimethyl borate, boric-acid triethyl, boric-acid tributyl, boric-acid trihexyl, boric-acid triheptyl, a boric-acid triphenyl, boric-acid tritolyl, boric-acid TORINAFUCHIRU, etc. are mentioned.

[0027] this invention -- concrete -- the (a) alkali metal compound and/or an alkaline-earth-metal compound, and a (b) nitrogen-containing basic compound -- a shell -- a catalyst is used preferably

[0028] Under the present circumstances, the (a) alkali metal compound and a /alkaline-earth-metal compound are used in the above-mentioned amount, and 10-6 to ten - one mol of (b) nitrogen-containing basic compounds is preferably used in the amount of 10-5 to ten - two mols to one mol of aromatic dihydroxy compounds. It is desirable at the point that the polycarbonate which an ester exchange reaction and polymerization reaction advanced at sufficient speed that the amount of the nitrogen-containing basic (above-mentioned b) compound was the above-mentioned range, and was further excellent in a hue, thermal resistance, water resistance, etc. is obtained.

[0029] Thus, the polycarbonate which the catalyst which combined the (a) alkali metal compound or the alkaline-earth-metal compound, and the (b) nitrogen-containing basic compound has high polymerization activity, can make generate the polycarbonate of the amount of macromolecules, and is moreover obtained is further excellent in thermal resistance and water

resistance, and the overtone tone is improved and it is excellent in transparency.

[0030] moreover -- this invention -- the (a) alkali metal compound and/or an alkaline-earth-metal compound, and the (c) boric acid or the ester of boric acid -- a shell -- a catalyst is used preferably and the catalyst which consists of the (a) alkali metal compound and/or an alkaline-earth-metal compound, a (b) nitrogen-containing basic compound, and the (c) boric acid or the ester of boric acid further is used preferably

[0031] As for such the (a) alkali metal compound or an alkaline-earth-metal compound, and a (b) nitrogen-containing basic compound, being used in an amount which was described above is desirable.

[0032] Moreover, the (c) boric acid or 10-7 to ten - two mols of 10-8 to ten - one mol of ester of boric acid are preferably used in the amount of 10-6 to ten - four mols still more preferably to one mol of aromatic dihydroxy compounds. (c) The amount of a boric acid or the ester of boric acid is desirable at the point that the polycarbonate to which the fall of the molecular weight after heat aging cannot take place that it is 10-8 to ten - one mol easily and which was further excellent in a hue, thermal resistance, and water resistance is obtained to one mol of aromatic dihydroxy compounds.

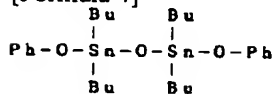
[0033] The polycarbonate which especially the catalyst that combined three persons who consist of the (a) alkali metal compound or an alkaline-earth-metal compound, a (b) nitrogen-containing basic compound, and the (c) boric acid or the ester of boric acid has high polymerization activity, can make generate the polycarbonate of the amount of macromolecules, and is moreover obtained is further excellent in thermal resistance and water resistance, and the overtone tone is improved further and is excellent in transparency.

[0034] Although the polycondensation reaction with a multifunctional compound can be performed the aromatic dihydroxy compound using such a catalyst, a carbonic acid diester, and if needed under the polycondensation reaction condition of the aromatic dihydroxy compound known conventionally and a carbonic acid diester, and the same conditions 80-250-degree C 100-230 degrees C are a 0 - 3-hour ordinary pressure still more preferably [ for 0 to 4 hours ] preferably [ it is still more desirable and / for 0 to 5 hours ] at the temperature of 120-190 degrees C preferably about the reaction of a first-stage eye, and both are made to specifically react. Subsequently, reaction temperature is raised, the reaction of an aromatic dihydroxy compound and a carbonic acid diester is performed, making the system of reaction reduced pressure, and, finally 5 or less mmHg of polycondensation reactions of an aromatic dihydroxy compound and a carbonic acid diester are preferably performed at the temperature of 240-320 degrees C under reduced pressure of 1 or less mmHg. Continuous system may perform the above polycondensation reactions and they may be performed by the batch type again. moreover -- even if the reactor used by facing for performing the above-mentioned reaction is a tub type and it is a juxtaductal type -- a column -- you may be type

[0035] The aromatic polycarbonate used for this invention can add an acid compound to the resultant obtained by doing in this way, i.e., a polycarbonate. As long as it can neutralize alkaline compounds used as a catalyst, such as an alkali metal compound and an alkaline-earth-metal compound, even if the above-mentioned acid compound is a Lewis-acid compound, it may be ester of the strong acid containing a Broensted-acid compound or a sulfur atom. Moreover, pKa in the inside of 25-degree C solution of a Broensted-acid compound is three or less especially preferably five or less. When pKa uses the acid compound in which such a value is shown, the alkali metal or alkaline earth metal used as a catalyst can be neutralized, and there is an advantage that the polycarbonate obtained can be stabilized.

[0036] As a Lewis-acid compound, specifically 2 (CH<sub>3</sub> COO) Z<sub>a</sub>, A boron compound, such as Sb 2O<sub>3</sub>, B(OPh) 3, boric-acid zinc, and phosphoric-acid boron, B(OCH<sub>3</sub>) 3, B(OEt) 3, and B(OPh) 3 etc. -- the ester of boric acid -- Aluminium compounds, such as an aluminum stearate and an aluminum silicate, Zirconium compounds, such as a carbonic-acid zirconium, an alkoxide zirconium, and a hydroxycarboxylic acid zirconium, Germanium compounds, such as gallium compounds, such as the Lynn-ized gallium and a gallium antimonide, a germanium dioxide, and organic germanium, a tetrapod, and HEKISAO luganot tin, [0037]

[Formula 4]



Titanium compounds, such as zinc compounds, such as bismuth compounds, such as antimony compounds, such as which tin compound, an antimony oxide, and alkyl antimony, a bismuth oxide, and an alkyl bismuth, and a zinc stearate, alkoxy titanium, and titanium oxide, etc. can be mentioned. In addition, in the inside of the above-mentioned formula, and Ph, a phenyl group and Et express an ethyl group and Bu expresses a butyl. As a Broensted-acid compound, moreover, specifically A phosphoric acid, phosphorous acid, hypophosphorous acid, a pyrophosphoric acid, polyphosphoric acid, a boric acid, A hydrochloric acid, a hydrobromic acid, a sulfuric acid, a sulfurous acid, an adipic acid, an azelaic acid, dodecane 12 acid, L ascorbic acid, an aspartic acid, a benzoic acid, a formic acid, an acetic acid, a citric acid, Glutamic acid, a salicylic acid, a nicotinic acid, a fumaric acid, a maleic acid, oxalic acid, A benzenesulfonic acid, a toluene sulfonic acid, and a benzenesulfonic acid, The compound of sulfonic acids, such as p-toluenesulfonic acid, a trifluoromethane sulfonic acid, a naphthalene sulfonic acid, sulfonated polystyrene, and a methyl-acrylate-sulfonation styrene copolymer, etc. can be mentioned.

[0038] As ester of the acid containing a sulfur atom, three or less compound is used for pKa of acid-residue portions, such as the methyl of a dimethyl sulfate, a diethyl sulfate, and p-toluenesulfonic acid, ethyl, butyl, an octyl or phenyl ester, a methyl of a benzenesulfonic acid, ethyl, butyl, an octyl, or phenyl ester.

[0039] The acid compound which contains a sulfur atom, the Lynn atom, etc. among such acid compounds is desirable, and the acid compound containing especially a sulfur atom is desirable. The acid compound added to a resultant is used by adding to the

polycarbonate obtained as a resultant in the amount which can neutralize or weaken influence with the alkaline compound which remains. For example, 0.1-100-mol 0.01-500 mols [ 0.1-50-mol ] are especially used in the amount of 0.5-30 mols preferably still more preferably to the alkali metal compound and/or one mol of alkaline-earth-metal compounds which remain in a polycarbonate.

[0040] When it is ester of the acid containing the Broensted acid or sulfur atom with which 0.01-500-mol 0.1-50 mols are preferably used in the amount of 0.1-30 mols still more preferably when especially an acid compound is the Broensted acid which has larger pKa than a Lewis acid or 3, and an acid compound has three or less pKa, 0.01-500-mol 0.1-15 mols are preferably used in the amount of 0.1-7 mols still more preferably.

[0041] The end hydroxy concentration of PC resin of this invention is determined by measuring the absorption intensity of 3600cm<sup>-1</sup> of FTIR. Total end group concentration is calculated by calculating a viscosity average molecular weight (M) from the following formulas based on the [eta] (limiting viscosity) value measured with the methylene-chloride solution (20 degrees C).

[0042] [Equation 1] the upper limit of the end hydroxyl concentration of PC resin used for manufacture of PC resin constituent of [eta] = 1.23x10<sup>-4</sup>xM<sup>0.83</sup> this invention -- 40-mol % -- desirable -- 30-mol % -- further -- desirable -- 20-mol % -- it is -- a minimum -- two-mol % -- it is five-mol % preferably if end hydroxyl concentration exceeds the above-mentioned upper limit, since the thermal stability of the PC resin itself manufactured will get worse, if it is under the above-mentioned minimum on the other hand, sufficient mold-release characteristic will obtain preferably -- not having -- moreover, metal mold -- since dirt is produced, it is not desirable

[0043] the molecular weight distribution (Mw/Mn) measured by the gel permeation chromatography of PC resin used by this invention -- an upper limit -- 2.8 -- it is 2.5 preferably and a minimum is 2.0 By putting in an end encapsulant (single organic-functions monomer), when making molecular weight distribution large, adjustment of molecular weight distribution can be performed by lengthening the residence time, in case it fuses at an elevated temperature on the other hand, when narrowing molecular weight distribution. Furthermore, in the case of a melting polymerization method, molecular weight distribution can be adjusted by adjusting the mole ratio of an aromatic dihydroxy compound and a carbonic acid diester, and molecular weight distribution show the inclination which becomes narrow because of the polymerization in an elevated temperature and a high vacuum.

[0044] if the above-mentioned partial ester is conventionally added to PC resin in amount sufficient for a mold-release characteristic -- the thermal stability of a resin constituent -- falling -- moreover, metal mold -- it was easy to generate dirt If partial ester is added to PC resin with the molecular weight distribution according to this invention, such a problem will be solved and a good mold-release characteristic and imprint nature will be attained.

[0045] The viscosity average molecular weights of PC resin used by this invention are 12,000-18,000 preferably. If the above-mentioned upper limit is exceeded, if a viscosity average molecular weight is under the above-mentioned minimum, mechanical strength is inadequate, and a fluidity is inadequate, and since it becomes easy to produce optical strain, it is not desirable.

[0046] In the above-mentioned PC resin 100 weight section, an upper limit can be acquired by the 0.08 weight section, and, as for PC resin constituent of this invention, a minimum can acquire preferably the partial ester of an aliphatic carboxylic acid and polyhydric alcohol 0.1 weights the 0.01 weight section and by adding in the amount of the 0.03 weight section preferably. When [than the above-mentioned upper limit] more, thermal stability has the fault of being bad, and when fewer than the above-mentioned minimum, it has the fault that the advanced imprint of sufficient mold-release characteristic and MIKURONO-DA - cannot be obtained.

[0047] Especially as the above-mentioned aliphatic carboxylic acid, it is not limited and both saturation and unsaturation aliphatic carboxylic acids can be used. For example, the hydrogenated animal oil can be used. As the above-mentioned aliphatic carboxylic acid, a saturation monovalent fatty acid is desirable and especially the thing of carbon numbers 12-24 is desirable. If there are few carbon numbers than the above-mentioned range, since the thermal stability of manufactured PC resin constituent will be inferior compared with the thing of the above-mentioned range and generating of gas will take place, it is not desirable. On the other hand, since the mold-release characteristic of PC resin constituent is inferior compared with the thing of above-mentioned within the limits when a carbon number is larger than the above-mentioned range, it is not desirable. Specifically as the above-mentioned aliphatic carboxylic acid, a dodecyl acid, a myristic acid, pentadecyl acid, a palmitic acid, a heptadecyl acid, stearin acid, a nonadecane acid, arachin acid, behenic acid, a lignoceric acid, etc. are mentioned.

[0048] Although it is not limited but all can use divalent, trivalent, tetravalence, pentavalence, 6 \*\*, etc. especially as the above-mentioned polyhydric alcohol, ethylene glycol, a glycerol, a trimethylol propane, a pentaerythritol, etc. are desirable, and especially a glycerol is desirable.

[0049] The partial ester of the aliphatic carboxylic acid and polyhydric alcohol which are used by this invention can be obtained by the esterification reaction of common use.

[0050] PC resin constituent of this invention can add the additive of common use, for example, a pigment, a color, reinforcing materials, a bulking agent, a heat-resistant stabilizer, an antioxidant, a weathering agent, an ultraviolet ray absorbent, lubricant, other release agents, a crystallizer, a plasticizer, a fluid improvement agent, an antistatic agent, an elastomer, a stabilizing agent, etc. according to the purpose at the time of fabrication at the time of kneading of a resin, unless the physical properties other than the above-mentioned component are spoiled.

[0051] As a heat-resistant stabilizer, a phenol system compound, the Lynn system compound, or a carboxylate can be mentioned.



[0052] As a phenol system compound, for example n-octadecyl-3-(4-hydroxy-3' and 5'-G t-buthylphenyl) propionate, Tetrakis [methylene-3-(3' and 5'-G t-butyl-4-hydroxyphenyl) propionate] methane, 1, 1, 3-tris (2-methyl-4-hydroxy-5-t-buthylphenyl) butane, distearyl (4-hydroxy-3-methyl-5-t-butyl) benzyl malonate, 4-hydroxymethyl -2, 6-G t-butylphenol, etc. are mentioned.

[0053] As a Lynn system compound, phosphoric ester, phosphorous acid ester, etc. are mentioned. As an example of phosphoric ester, trimethyl phosphate, triethyl phosphate, Tributyl phosphate, trioctylphosphate, tridecyl phosphate, Trio KUTADE sill phosphate, distearyl pentaerythrityl diphosphate, Tricyclo alkyl phosphate, such as trialkyl phosphate; tricyclohexyl phosphate, such as tris (2-chloro ethyl) phosphate and tris (2, 3-dichloro propyl) phosphate; Triphenyl phosphate, tricresyl phosphate, Triaryl phosphate, such as tris (nonylphenyl) phosphate and 2-ethyl phenyl diphenyl phosphate, etc. is mentioned. As an example of phosphorous acid ester, trimethyl phosphite, triethyl phosphite, Tributyl phosphite, trioctyl phosphite, tris (2-ethylhexyl) phosphite, TORINO nil phosphite, tridecyl phosphite, trio KUTADE sill phosphite, Tristearylphosphite, tris (2-chloro ethyl) phosphite, trialkyl phosphite [ , such as tris (2, 3-dichloro propyl) phosphite, ] ; -- tricyclo alkyl phosphite [ , such as tricyclohexyl phosphite ] ; -- triphenyl phosphite -- Tricresyl phosphite, tris (ethyl phenyl) phosphite, Tris (2, 4-G t-buthylphenyl) phosphite, tris (nonylphenyl) phosphite, Triaryl phosphite, such as tris (hydroxyphenyl) phosphite; Phenyl JDESHIRU phosphite, Diphenyl desyl phosphite, diphenyl isooctylphosphite, Further Arylated-alkyl phosphite, such as phenyl isooctylphosphite and 2-ethylhexyl diphenyl phosphite; Distearyl pentaerythrityl diphosphate, General formula P(OR) 3, such as bis(2,4-di-tert-butylphenyl) pen TAECHISU retail diphosphate (here, R is an aliphatic hydrocarbon machine, an alicycle group hydrocarbon group, or an aromatic-hydrocarbon machine independently, respectively.) The phosphorous acid ester which can be expressed is mentioned. Or you may use hypophosphorous acid, a pyrophosphoric acid, polyphosphoric acid, etc. These also use phosphorous acid ester, especially tris (2, 4-G t-buthylphenyl) phosphite more preferably especially. As an example of a carboxylate, although n-octadecyl -3 (4'-hydroxy-3' and 5'-G t-buthylphenyl) propionate, various ant cyclic diepoxy carboxylate, etc. can be mentioned, it is not limited to these.

[0054] Even if it uses the above-mentioned heat-resistant stabilizer independently, two or more sorts may be mixed and used for it. As for these compounds, it is desirable to be used in the amount below the 0.1 weight sections to the polycarbonate 100 weight section.

[0055] Other additives are more specifically indicated by JP,4-175368,A.

[0056] The method for manufacturing the resin constituent of this invention should just be the melting kneading method. Although use of a little solvent is also possible, generally it is unnecessary. An extruder, a BA@BBARI mixer, a roller, a kneader, etc. can be mentioned especially as equipment, and these are operated batch-wise or continuously. Especially the order of mixture of a component is not restricted.

[0057] PC resin constituent of this invention is excellent in thermal stability, a mold-release characteristic, and the advanced imprint nature of micron order, and has the effect of being hard to produce the dirt of metal mold at the time of continuous molding. Since the molecular weight distribution of PC resin (1) Used are narrow and there are few low-molecular-weight objects, this Even if it adds a low-molecular-weight object like partial ester comparatively so much, aggravation of thermal stability is not caused. Moreover, it thinks because the end hydroxyl of PC resin which caused and (2) Used the dirt of metal mold at the time of continuous molding, and the synergistic effect of partial ester show the mold-release characteristic excellent also in addition of a small amount of partial ester, and the advanced imprint nature of micron order.

[0058]

[Example] Below, an example explains this invention still in detail. The "section" is the weight section. In addition, in the following examples and examples of comparison, the following PC resin and the ester of an aliphatic carboxylic acid and polyhydric alcohol were used.

(A) PC resin PC (1) : the 0.44K (Japanese GE plastics company make) mol of bisphenol A for examples and the 0.46K (product made from ENII) mol of JIFENIRUKA-BONETO were taught to the 250l. tub type mixing vessel, and after carrying out a nitrogen purge, it dissolved at 140 degrees C. Next, to 180 degrees C, the temperature up of this was carried out and it was agitated for 30 more minutes. Next, 0.11 mols and 0.00044 mols of sodium hydroxides were added for tetramethylammonium hydroxide as a catalyst, and while raising temperature to 240 degrees C, the pressure was gradually lowered to 30mmHg(s). The amount of the phenol which keeps thermal stress constant and distills it was measured, and when the phenol to distill was lost, it returned to atmospheric pressure with nitrogen. The time which the reaction took was 2 hours. The ultimate clay [eta] of the obtained reactant was 0.12 dl/g.

[0059] Next, the pressure up of this reactant was carried out with the gear pump, it fed into the centrifugal type thin film evaporation machine, and the reaction was advanced. The temperature of a thin film evaporation machine and the pressure were controlled to 270 degrees C and 2mmHg, respectively. From the evaporation machine lower part, it sent into the biaxial horizontal-type churning polymerization tank controlled by 270 degrees C and 0.2mmHg with the gear pump in 40kg /in an hour, and the polymerization was carried out in [ residence-time ] 30 minutes. The viscosity average molecular weight of the obtained polymer is 15500, and end hydroxyl concentration is 15-mol %. The molecular weight distribution by GPC measurement were 2.2.

PC (2): The sodium hydroxide was performed like manufacture of PC (1) using the 0.44K mol of bisphenol A for the examples of comparison, and the 0.48K mol of JIFENIRUKA-BONE-TO except 0.00011 mols having added. The time which the reaction took was 4 hours. Next, the processing same to this reactant as PC (1) was performed. 15500 and the end hydroxyl concentration of the viscosity average molecular weight of the obtained polymer were three-mol %. The molecular weight distribution by GPC measurement were 3.0.



- [0060] PC (3): It carried out like manufacture of PC (1) using the 0.44K mol of bisphenol A for the examples of comparison, and the 0.443K mol of JIFENIRUKA-BONE-TO. 15500 and the end hydroxyl concentration of the viscosity average molecular weight of the obtained polymer were 50-mol %. The molecular weight distribution by GPC measurement were 2.2.
- [0061] PC (4): The polymerization was carried out to bisphenol A for the examples of comparison by the method of common use using the phosgene, and end closure was carried out using the PARAKU mill phenol like common use. Molecular weight distribution according [ 15500 and end hydroxyl concentration ] to zero-mol measurement [ % and GPC ] in the viscosity average molecular weight of the obtained polymer were 3.0.
- [0062] The end hydroxyl concentration, the molecular weight distribution (Mw/Mn), and the viscosity average molecular weight of obtained PC resin are collectively shown in Table 1.
- [0063]  
[Table 1]

表 1

PC樹脂	製 法	末端OH濃度 (モル%)	分子重分布 (Mw/Mn)	粘度平均分子量
PC (1)	熔融法	15	2.2	15500
PC (2)	熔融法	15	3.0	15500
PC (3)	熔融法	50	2.2	15500
PC (4)	界面法	0	3.0	15500

(B) Ester SMG of an aliphatic carboxylic acid and polyhydric alcohol : stearin acid monoglyceride (for examples)  
STG: Stearin acid triglyceride (for the examples of comparison)

PETS: Pentaerythritol tetrapod stearate (for the examples of comparison).

[0064] Melting kneading of the component of the rate (weight ratio) shown in examples 1-3 and the example 1 of comparison - the 7 following table 2 was carried out at the setting temperature of 260 degrees C using the 1 shaft extruder (ratio-of-length-to-diameter=17.5), and the pellet was created. The following evaluations were performed about the obtained pellet.

(1) Using the stamper made from mold-release characteristic nickel, the compact disk substrate of phi 120 was fabricated 10,000 shots of continuation on 350 degrees C of cylinder temperatures, the die temperature of 80 degrees C, and the conditions for 1 cycle 7 seconds, and the remainder to a disk or the metal mold of a sprue was evaluated by CD making machine as a percent defective (%) on them.

(2) The pit imprinted by the disk substrate obtained by the method of imprint nature (1) was observed and evaluated with the optical microscope (800 times). Ten disk substrates were evaluated about each of an example and the example of comparison.

(3) The method of the dirt (1) of a stamper observed and estimated the metal mold after 10,000-shot fabrication, and the state of a stamper by the eye.

(4) After stopping fabrication for 15 minutes and making a resin pile up by the process condition of thermal stability (1), fabrication was started again and the hue and surface state of a disk substrate which were obtained were evaluated. The surface state was observed visually. A hue (YI) is Color and Color Defference Meter ND-1001 DP made from Nippon Denshoku Industry about X, Y, and Z value. It uses, measures with a transmission method and is about the degree of yellow (YI). [0065]

[Equation 2]

It computed in  $YI = (1.277X - 1.060Z) \times 100 / Y$ . A result is shown in Table 3. In a table, "silver" is the abbreviation for a silver streak.

[0066]

[Table 3]

表 3

	実 施 例			比 較 例						
	1	2	3	1	2	3	4	5	6	7
評価項目										
成型性 [不良率 (%) ]	2.0	1.0	1.0	連続生産不可	1.5	2.2	1.5	1.0	2.5	3.5
転写性	すべて	すべて	すべて	すべて	すべて	すべて	すべて	4枚が	すべて	すべて
(ビット形状)	良好 (鮮明)	良好 (鮮明)	良好 (鮮明)	形状にムラ有 (不鮮明)	良好 (鮮明)	良好 (鮮明)	良好 (鮮明)	不鮮明 (再現乏しい)	形状にムラ有 (不鮮明)	形状にムラ有 (不鮮明)
スタンパー汚れ	少ない	少ない	少ない	—	外周部に汚れ目立つ	少ない	外周部に汚れ目立つ	外周～内周にかけて汚れ多い	少ない	少ない
熱安定性										
色相 (YI)	2.1	2.3	2.3	1.9	2.6	2.7	2.8	2.6	2.1	2.2
表面状態	良好	良好	良好	良好	シルバー発生	シルバー発生	シルバーヤケ発生	シルバーヤケ発生	良好	良好

PC resin constituent of this invention showed the property which was excellent in all evaluation criteria. PC resin constituent which consists of the aliphatic monoglyceride of an amount enough for the conventional PC resin and a mold-release characteristic so that clearly from the examples 2-4 of comparison -- thermal stability -- bad -- metal mold -- dirt arose In the examples 6 and 7 of comparison using full ester, a mold-release characteristic and imprint nature are inferior.

[0067]

[Effect of the Invention] PC resin constituent of this invention is excellent in thermal stability, it is hard to produce the dirt of metal mold at the time of continuous molding, and it is further excellent in a mold-release characteristic, and also fits the advanced imprint of the micron order demanded in manufacture of an optical disk. So, it is very useful as a resin constituent for optics.

[Table 2]

表 2

	実 施 例			比 較 例						
	1	2	3	1	2	3	4	5	6	7
PC(1)	100	100	100	100	-	-	-	100	100	100
PC(2)	-	-	-	-	100	-	-	-	-	-
PC(3)	-	-	-	-	-	100	-	-	-	-
PC(4)	-	-	-	-	-	-	100	-	-	-
SMG	0.01	0.05	0.1	-	0.05	0.05	0.05	0.3	-	-
STG	-	-	-	-	-	-	-	-	0.05	-
PETS	-	-	-	-	-	-	-	-	-	0.05

[Translation done.]